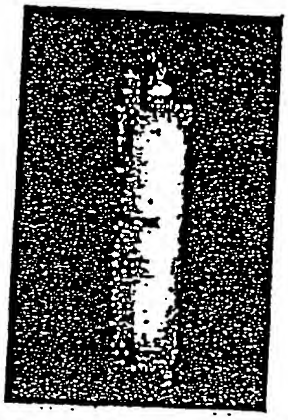
Appendix H

Product literature from Millipore Corporation

Planargard Disposable Filters



Planargard Disposable filters offer convenience and versatility when filtering CMP slurries, with full compatibility for both oxide and metal slurries. The unique design, employing standard Flaretek fittings and a fully integrated polypropylene filter and shell, provides automatic draining capabilities, reduced holdup volume, and minimized exposure during filter change-out. Planargard Disposable filters avoid problems caused by non-standard fittings , and allow users to choose a range of options from maximum filter life to fewer wafer defects. Planargard Disposable filters — the result of Millipore's extensive applications expertise — represent the latest advances in proven filtration capabilities.

Specifications

Materials of Construction

All polypropylene construction with no O-rings or elastomeric seals.

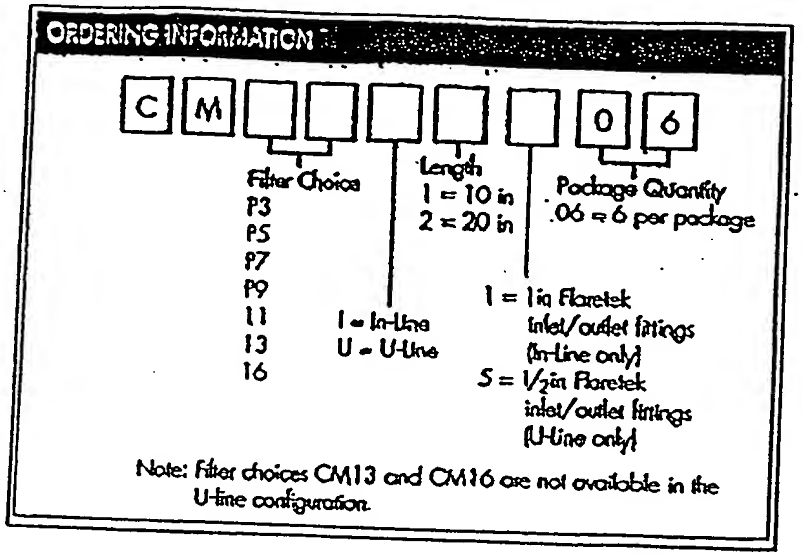
Connections

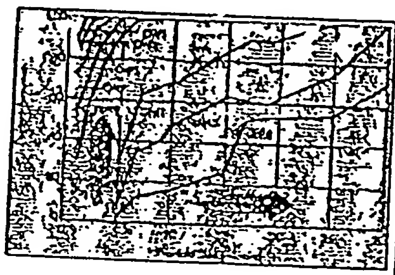
Inlet/Outlet
1/2 in Flaretek fittings, 1 in Flaretek fittings

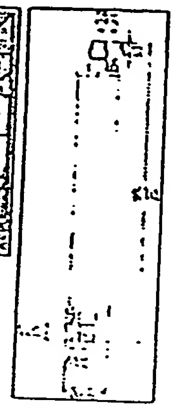
Vent/Drain
1/4 in Flaretek fittings

Operating Conditions

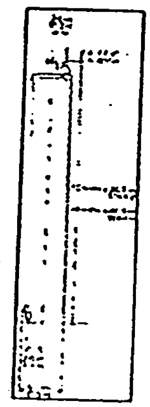
Maximum Pressure
4.1 bar (60 psi) at 25 °C







Planargard disposable In-Line filter with 1 in Flaretek inlet/outlet fittings and 1/4 in Flaretek vent/drain fittings



Planargard disposable U-Line filter with 1/2 in Flaretek inlet/outlet fittings and 1/4 in Flaretek drain fittings

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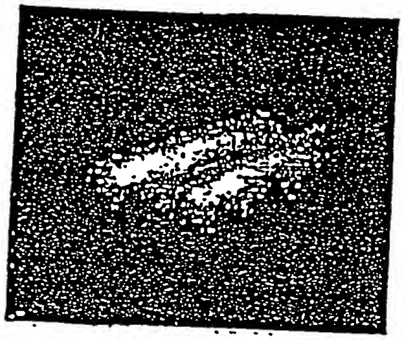
Millipore Planargard Filters



Description

- Graded-density filter removes agglomerated large particles before they can scratch wafer surfaces
- Removes disruptive particles without removing small working particles, ensuring consistent slurry delivery to your process
- All polypropylene construction provides excellent compatibility with high and low pH slurries

Applications

- Point-of-use and bulk filtration of ILD slurries for chemical mechanical planarization (CMP)

Specifications

Materials

All-polypropylene construction
O-rings
EPR

Cartridge Dimensions

Diameter
70 mm (2.75 in)
Length
10": 264 mm (10.4 in)
20": 512 mm (20.2 in)
30": 761 mm (30 in)

Connections

Code 0 2-222 double O-ring

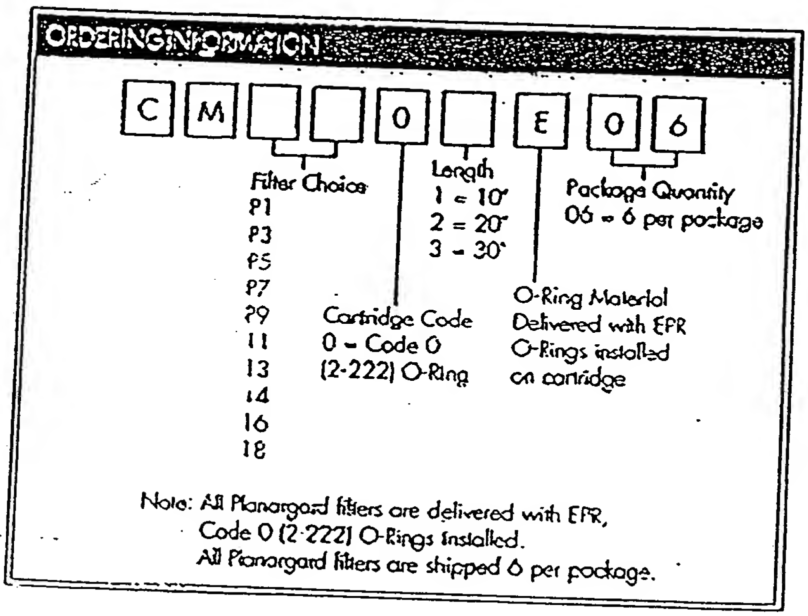
Operating Conditions

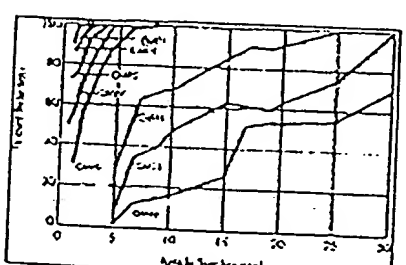
Maximum Forward Differential Pressure

4.8 bar (70 psi) at 20 °C

Filter Selection Guide

Filtration Objective	Location	Suggested Product
Minimize wafer defects	Point of use	CMP1, CMP3, CMP5
Extend filter life	Point of use	CMP7, CMP9, CM11
Remove gross contaminants	Point of use	CM13
Reduce large particles formed in shipping container	Intake	CM14, CM16, CM18
Reduce large particles and gels formed during dilution	Post-dilution	CMP9, CM11, CM13
Continuous cleaning	Recirculation loop	CM14, CM16, CM18





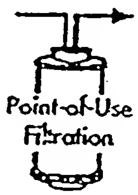
Actual Particle Retention in Oxide SlurryXXX

For a complete technical data sheet, click here.

Guide to Millipore Products for CMP Processes

The CMP slurry filtration challenge is to remove large particles and agglomerates from slurry that can cause defects, without changing slurry performance.

Point-of-Use Filtration



CMP Slurries introduce millions of abrasive particles to the wafer, potentially causing scratches on the surface. Point-of-use filtration is the most effective location for reducing the number of defect-causing particles. We have seen up to a ten-fold reduction in light point defects by using Planargard filters at the point-of-use.

For more applications information, see our Millipore technical document, MA071 POU <u>Filtration of Silica-Based CMP Slurries Using Planargard Filters</u> by Zhenwu Lin, Joseph Zahka, Geanne Vasilopoulos.

Point of Use Filtration Selection Guide

Find the type of slurry used in your process and select the filter for specific product information.

Slurry Type	Recommended Filter

Fumed Silica Oxide (Diluted Cabot[R] SS25 and SC1, SS12, SC112; Rodel[R] ILD1300, ILD1200)	o Planargard Cartridge Filter, membrane type CMP5 o Planargard Disposable Filter, membrane type CMP5 o Planargard Cartridge Filter, membrane type CMP3 o Planargard Disposable Filter, membrane type CMP3
Colloidal Silica Oxide (Klebosol[R] slurries)	o Planargard Cartridge Filter, membrane type CMP5 o Planargard Disposable Filter, membrane type CMP5 o Planargard Cartridge Filter, membrane type CMP3 o Planargard Disposable Filter, membrane type CMP3
Silica-based Metal (Cabot SSW200)	o Planargard Cartridge Filter, membrane type CMP5 o Planargard Disposable Filter, membrane type CMP5
Metal (non-silica based) <2% solids >2% solids	Please contact Millipore Technical Service

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POU Filtration of Silica-Based CMP Slurries Using Planargard Filters

Zhenwu Lin, Joseph Zahka, Geanne Vasilopoulos

Introduction

Chemical Mechanical Polishing (CMP) has become an enabling technology in semiconductor device manufacturing. The CMP process uses submicron (30 - 200 nm) silica slurries at a typical concentration of 10-30% solids. Typical silica slurries can contain a small number (104 to 106 counts/ml) of > 1.0 micron particles, which could potentially cause defects (microscratches) on the planarized wafer surfaces. The slurry solution presents unique challenges in delivery, filtration, and particle measurement.

The silica slurries used in the CMP process are stabilized suspensions of fine particles, typically 30 to 200 nm in size with concentrations ranging from 10 to 30%, in aqueous solutions with a specific pH. These slurries, which may need on-site dilution in the FABs, are applied directly onto the rotating pad to polish wafers and achieve global planarity. Fumed silica-based and "colloidal" silica-based slurries are the two families that are commercially available.

Fumed silica slurry is manufactured in two steps: 1) production of fumed silica (three-dimensional branched chain aggregates) by the vapor phase hydrolysis of silicon tetrachloride in a hydrogen oxygen flame [2]; 2) dispersion of fumed silica in aqueous medium with certain additives. Commercial fumed silica CMP slurries normally contain silica aggregates with mean particle sizes ranging from 100 nm to 200 nm. Figure 1a shows a SEM picture of typical silica aggregates.

"Colloidal" silica is produced from a dilute aqueous solution of water glass through deionization/nucleation, polymerization, particle growth and concentration steps [3]. All process steps are in the liquid phase. The silica particles formed are normally spherical. Commercial colloidal silica CMP slurries have particles with mean sizes ranging from 30 nm to 50 nm. Figure 1b shows a SEM picture of typical colloidal silica particles.

Filtration Needs

The typical specification for commercial silica slurries includes percent solids, pH, specific gravity, mean particle size and general (bulk) particle size distribution. However, a small number of "large" particles (>1m) have been found which fall outside of the specified size distribution. These particles, which can be aggregates, agglomerates. SEM images shown in Figures 2A and 2B confirm their existence. These large particles may come from agglomeration or local drying of slurry on shipping containers and in the distribution system. Gels may form due to pH shocks during dilution or

temperature fluctuations during shipment and storage.

There is no definitive information available on what size or type of particles can cause microscratches and particle contamination on wafer surfaces. However, higher numbers of "large particles" have been found to cause higher incidence of microscratches and particle contamination on polished wafers. Slurry filtration has proven to be beneficial in reducing wafer defects and increasing yields in CMP processes[1].

A control oxide CMP polishing experiment was conducted using a commercial fumed silica slurry contaminated with 5 m silica particles (~ 104 particles/ml). The polishing were performed on an IPEC/Westech 472 tool using a standard oxide CMP recipe. The bare wafers were deposited with 1000 nm PECVD SiO2 before polishing. Figure 3 and 4 show the surface scan results on the wafers polished with and without point-of-use filtration. A ten-fold reduction of light point defects was achieved by using a Planargard CMP5 filter at the POU.

Slurry Characterization

The main challenge of slurry filtration is to selectively retain the small number of defect-causing "large" particles (i.e., 104 to 106 counts/ml greater than 1 um) without retaining the desirable, small particles (30 to 200 nm) present in very high concentration (> 1015 counts/ml). There should be no measurable changes to the slurry's percent solids concentration and bulk particle size distribution before and after filtration. Therefore, filters to be used in CMP slurry filtration should be evaluated *in slurry* to validate their performance for the following attributes:

- Retention efficiency for "large" particles
- % solids content and bulk particle size distribution before and after filtration
- Throughput (Lifetime)

1. Detection of "Large" Particles

Quantitative determination of "large particles" is required to determine filter retention and a correlation between large particle concentration and wafer defects. There is no commercial particle counters available that can be used to detect the large particles (104 to 106 counts/ml >1 um) in the presence of bulk slurry particles (> 1015 counts/ml), without substantial sample dilution. Various particle counters were evaluated to determine their ability to detect the large particles with maximum tolerance to high concentrations of small particles (minimum sample dilution) and with ease of operation. A light scattering instrument was selected for the slurry application.

A typical schematic of the particle counting system, shown in Figure 5, includes continuous on-line dilution of slurry. The dilution factor should be high enough to minimize the interference caused by small particles in the slurry. Figure 6 shows a typical profile of large particle concentrations in oxide slurry before and after filtration. Filter retention for particles of a specific size can then be calculated based on the particle concentration before and after filtration.

2. Measurement of Bulk Particle Size Distribution and Percent Solids

Bulk particle size distribution (PSD) can be measured by many techniques [4]. The two most commonly used techniques in CMP slurries are light scattering and chromatography (i.e., capillary hydrodynamic fractionation, or CHDF). The light scattering instrument used for slurry PSD measurement is based on photon correlation spectroscopy (PCS), also referred to as quasi-elastic light scattering (QELS) or time-dependent light-scattering. With PCS, the size information is obtained from the time dependent fluctuation of scattered intensity due to concentration fluctuations resulting from Brownian motion of particles[4].

CHDF is based on the size exclusion effects that occur when a dispersion of particles flows through a

capillary tube. Laminar flow in the capillary tube has a parabolic velocity profile. Smaller particles can reach the slower streamline close to the tube wall due to Brownian motion, while larger particles cannot. Therefore, large particles exit the capillary tube faster than smaller ones. CHDF can be used to measure particles between 15 nm and 1 micron with up to 1 % solids [5]. This method was used for slurry particle size distribution measurement.

Another PSD instrument is based on acoustic attenuation spectroscopy. When acoustic waves propagate through a medium with suspended particles, the acoustic beam will be further attenuated by the particles by a variety of mechanisms. The acoustic attenuation spectrum can be detected and then inverted to obtain a mean particle size, a particle size distribution, and a dispersion concentration.

The percent solids in slurry can be calculated by drying a slurry sample of known weight.

Performance of Planargard TM Filters

Filter performance should be evaluated in slurry to determine: (a) retention efficiency for large particles; (b) percent solids content and bulk particle size distribution before and after filtration; (c) filtration throughput. This information is necessary for CMP process engineers to implement proper filtration.

Conventional microporous membrane filters will not work due to the high solids concentration in the slurry that forms a cake and plugs membrane quickly. Graded-density non-woven depth filters are preferred for this application. Filtration experiments were conducted with all-polypropylene graded-density PlanargardTM filters using silica slurry in a single-pass configuration to simulate point-of-use applications. Feed and filtrate samples were taken and analyzed for large particle concentration, percent solids, and bulk particle size distribution. The filter retention efficiency is defined as:

$$\text{Retention} = \frac{\text{Concentration in feed} - \text{Concentration in filtrate}}{\text{Concentration in feed}} \times 100\%$$

The retention efficiency curves for various Planargard filters are shown in Figure 7.

Throughput was measured based on the differential pressure across the filter as a function of filtered volume. A typical plugging curve is illustrated in Figure 8, which shows a gradual increase in differential pressure across the filter. The differential pressure increases slowly initially, but then climbs rapidly as the filter reaches the end of life. Data analysis proves that the plugging process follows the complete plugging mechanism, which can be represented by a linear relationship between the inverse differential pressure across the filter (or DPmin/DP in dimensionless form) and filtration volume. In this mechanism, the pressure drop across the filter increases slowly at the beginning, which will then increase exponentially. The importance of understanding the plugging process is to determine the filter change out time before the pressure drop reaches the region of exponential increase.

To maintain process control the filter should not affect the slurry's composition and the filter should have consistent retention throughout its useful lifetime. Figure 9 shows that filter retention remains fairly constant throughout its lifetime. Table I shows that the filter does not change the percent solids and mean particle size in the slurry, which is extremely important for a CMP process. As long as the solids concentration is not changed before and after filtration, the filter will not alter the bulk particle size distribution. Figure 10 shows the slurry's bulk particle size distribution in the feed and filtrates at 60% and 95% of the CMP5 filter's throughput.

Implementation Strategy

Implementation of silica slurry filtration depends on CMP process requirements, space availability, and the characteristics of the slurries. A number of field tests have demonstrated that point-of-use (POU) filtration at the tool can provide the most benefit in wafer defect reduction. Table II lists the normalized test results for oxide CMP with POU filtration.

Other filtration locations are at post-dilution, in the distribution loop, and at the slurry supply drum/tote.

It is strongly recommended that the implementation should start at POU filtration with higher retention filters to realize the maximum defect reduction benefits during CMP process development and qualification. To optimize the process, filtration at other locations can be used to supplement POU filter(s) and potentially to extend the life of POU filters.

Conclusion

"Large particles" have been detected in CMP slurries using an optical particle counter. SEM evaluation of slurry particles on membrane filters confirmed their existence. These defect-causing large particles may come from agglomeration, local drying of slurry on shipping containers and in the distribution system, and gel formation due to pH shocks during dilution and temperature fluctuations.

Graded-density depth filter can be used effectively to remove the defect-causing large particles without measurable change to slurry composition. Field test data have demonstrated the benefits of filtration on wafer defect reduction during CMP processes.

The optimal strategy to implement filtration in CMP processes can be dependent on site, process, and slurry type. POU filtration with higher retention filters is recommended to realize maximum defect reduction benefits during CMP process development and qualification.

References

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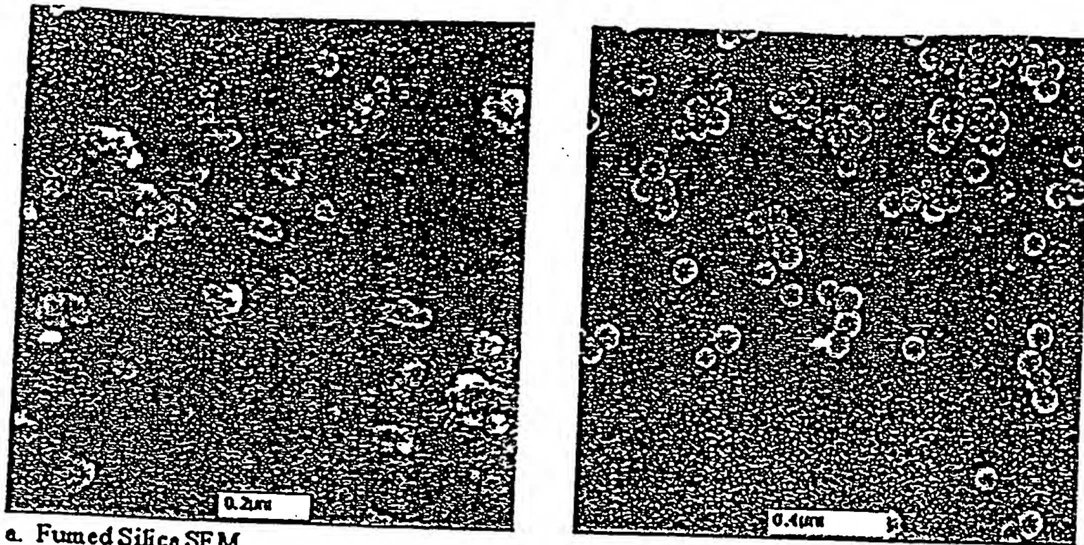
5. J.G. DosRamos and C.A. Silebi, "Size Analysis of simple and complex mixtures of colloids in the submicrometer ranges using capillary hydrodynamic fractionation", Chapter 19, ACS Symposium Series 472, 1990
6. Iler, R. K., The Chemistry of Silica, John Weily & Sons, New York, NY, 1979

Figure 1

Fumed Silica Aggregates and Colloidal Silica Particles



a. Fumed Silica SEM

b. Colloidal Silica SEM

Figure 2
"Large Particles" and Gel in Silica Slurries

2A

2B

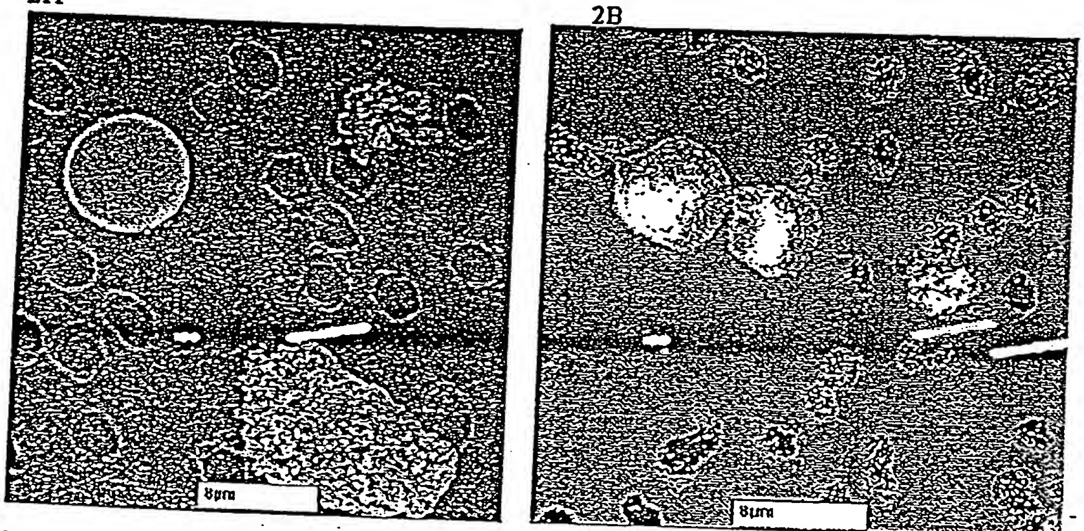


Figure 3 Wafer Scan Result Using Tencor SURFSCAN 6400 for the Wafer Polished with Filtered Slurry

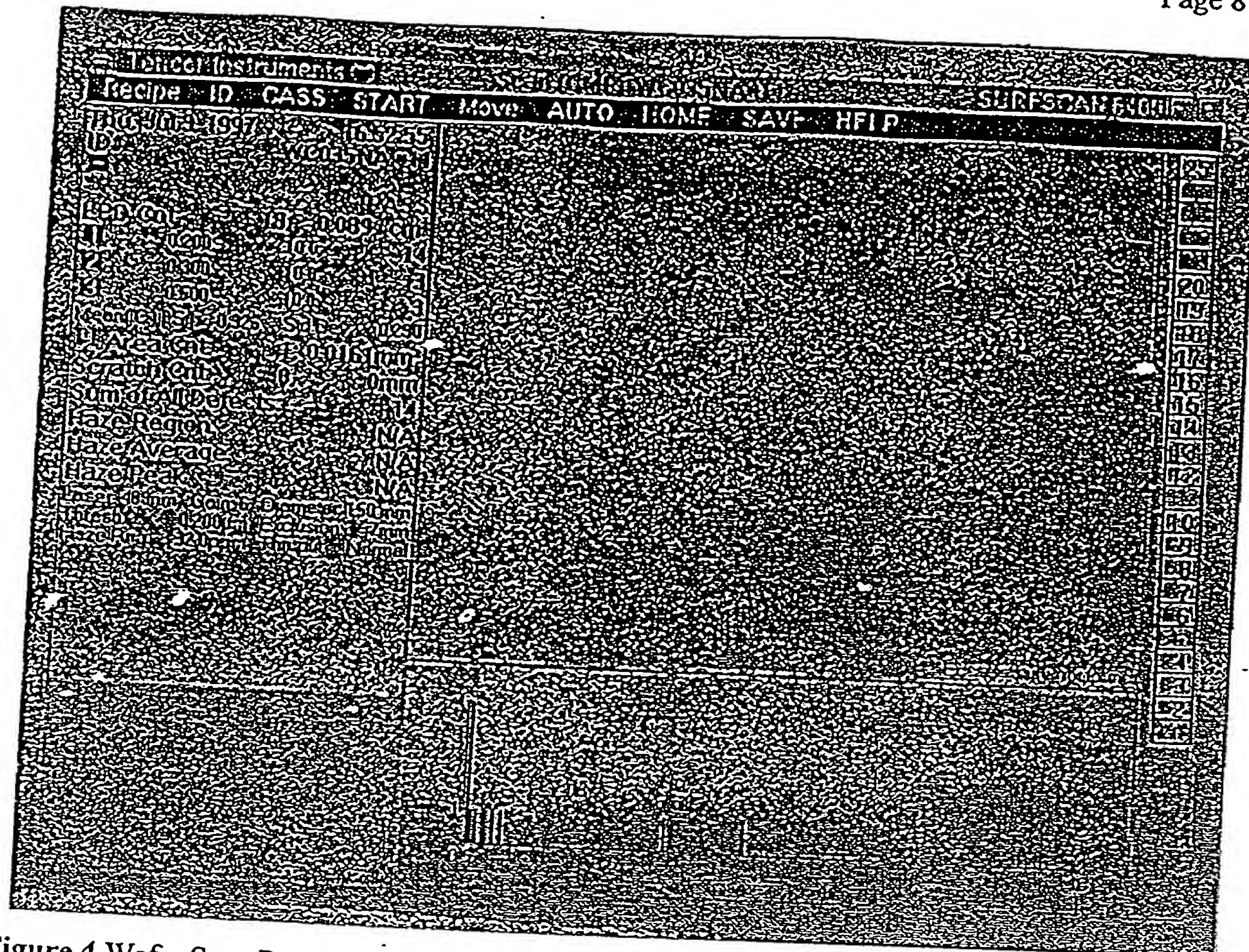


Figure 4 Wafer Scan Result Using Tencor SURFSCAN 6400 for the Wafer Polished with UN-filtered Slurry

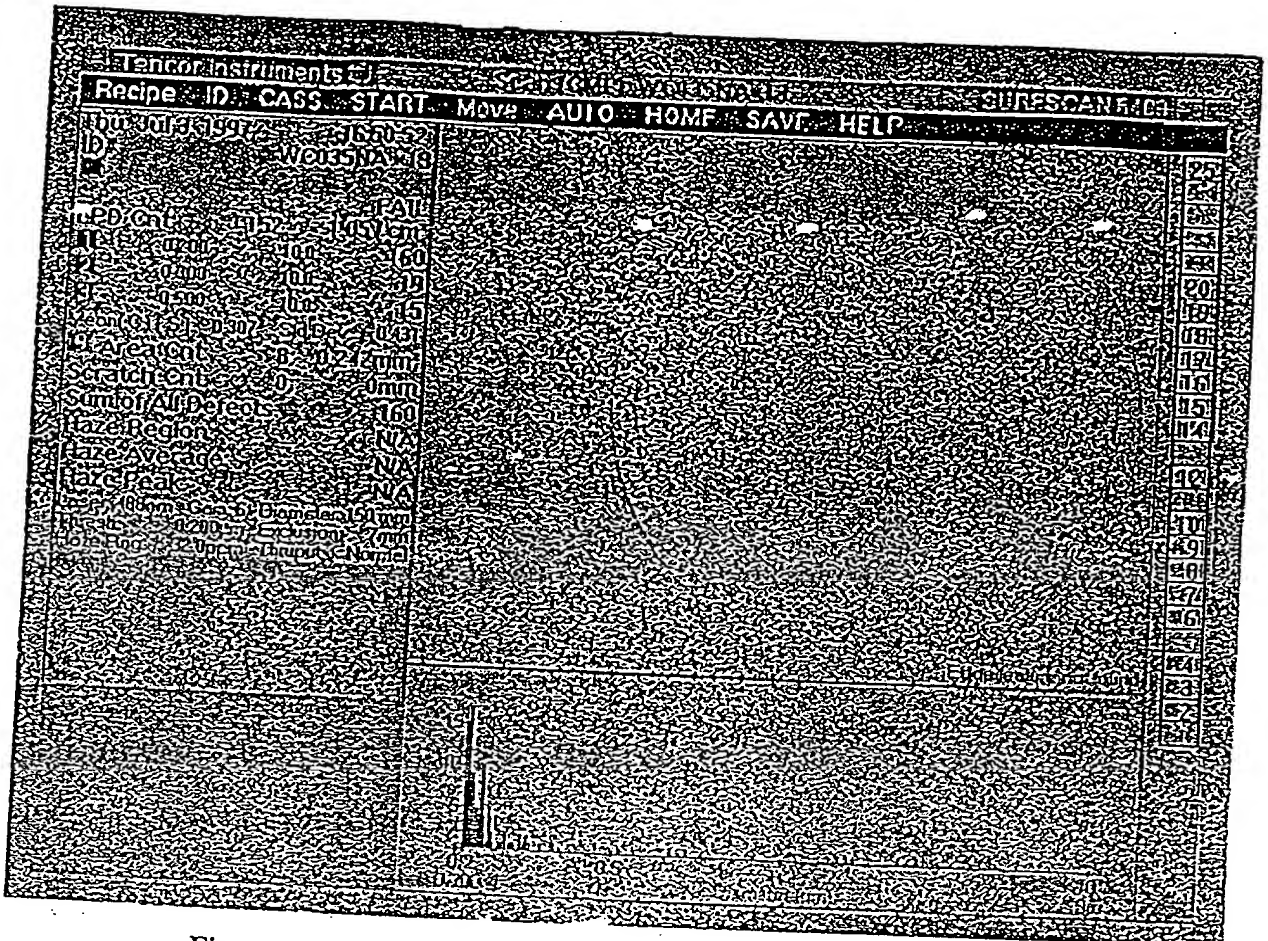


Figure 5

Figure 6

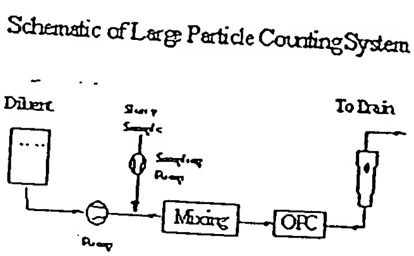


Figure 7
Retention Efficiency of POU
Planargard TM Filters

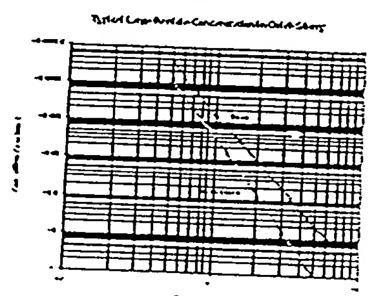


Figure 8
CMP5 Filter Plugging Curve
in Typical Silica Slurry

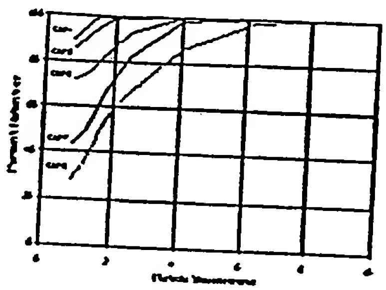


Figure 9
Retention Efficiency and Filter Life

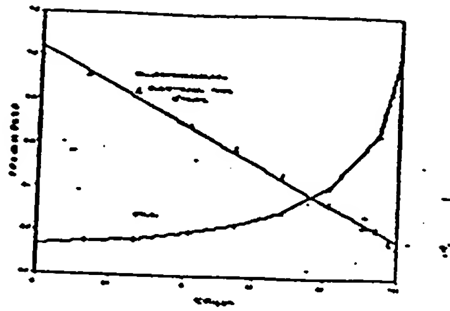
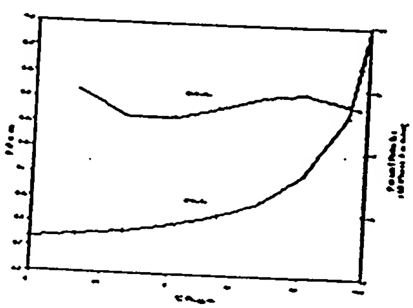


Figure 10
Effect on CMP5 Filter on Slurry Bulk Particle Size Distribution



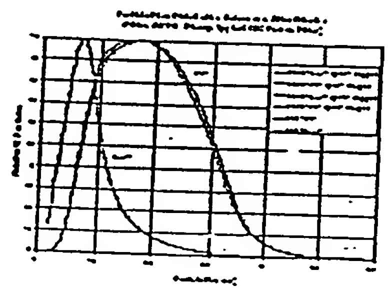


Table I
Effect of Filter Plugging on
Percent Solids and Mean Particle Size in Slurry

	Feed	60% Plugged	95% Plugged
%Solids	12.6	12.4	12.5
DW (nm)	192.3	197.	197.1
DW (nm)	93.3	93.3	94.1

DW: Mean diameter by weight
DN: Mean diameter by number

Table II Field Test Results
Light Point Defect (LDP) Reduction by POU Slurry Filtration

Customer	Slurry	Filters Tested at POU	Normalized LPD Levels
A	Fumed Silica	None CMP+CMP3	100 10
B	Colloidal Silica	None CMP7+CMP5 CMP3+CMP1	100 30 9
C	Colloidal Silica	None CMP3	100 33

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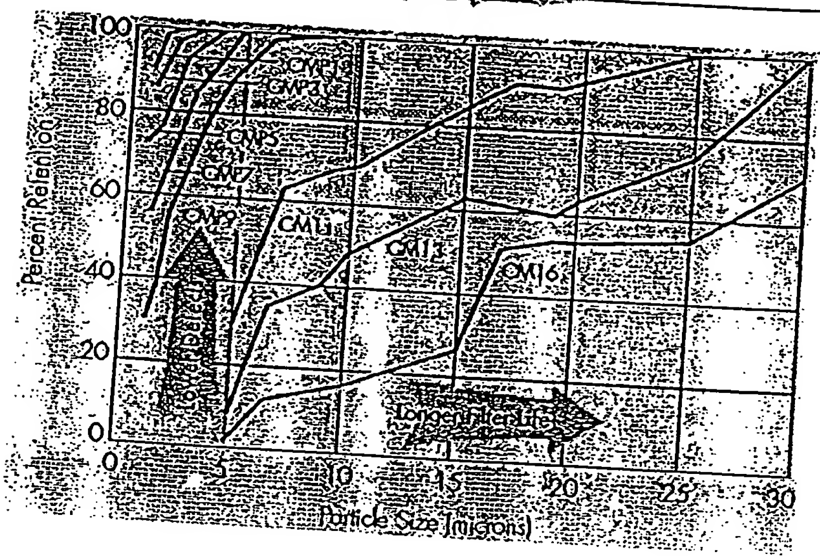
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100
80
60
40
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0
CMP5
CM11
CM13
CM16
Longer Filter Life
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